

The effectiveness of Henna as corrosion inhibitor for lead of battery in the sulphuric acid medium

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ABSTRACT/RESUME

Abstract: The study carried out is based on the improvement of lead-acid battery behaviour, protecting lead from attacks of 5M sulphuric acids. The conservation of its properties is guaranteed thanks to the Henna green inhibitor. Our hypothesis is reinforced by electrochemical results: resistance to the acidity of sulphuric acids and significant inhibition by adsorbing to the lead surface according to the Langmuir isotherm. One of the big enemy factors of lead-acid batteries is temperature, when it increases, it damages its capacity; but our surfactant at a temperature of 339 K produced a 118 Ohm.cm² corrosion resistance with enthalpy $\Delta H_{inh} = 4.20 \text{ KJ.mol}^{-1}$ using only 0.08 ml in 25ml (H₂SO₄5M). Thus, we can see the effectiveness of the henna extract; the protective power increases with the increase of the temperature.

I. Introduction

The storage of electrical energy is now widespread and commonplace in many electronic applications. The technologies that dominate to satisfy these needs are the electric batteries. [1] It represents a significant fraction of lead (Pb) use in Western Europe, almost 65-70%, for lead accumulators.

The lead-acid battery has indeed the double disadvantage of a mass energy, one of the weakest batteries 35Wh / kg, of a limited number of cycles, and also sensitive to the heat and bad conditions can reduce the number of cycles to less than one hundred; their principle is so simple that they can hardly be improved.

The accumulator is one of the most important subjects [2-3] for the most treated because of the low cost of manufacture. Each researcher chooses his axis of study by looking at the positive electrodes PbO₂ and negative Pb [4] by adding them substances, [5-7] in the grid [8], the paste; or adding the inhibitor to electrolyte. All of them lead to one goal which is to improve the battery

behaviour as well as to increase the lifetime. [9] And with this process we can avoid the accumulation of unusable battery waste and gain costs [10-12].

Our work consists in increasing the corrosion resistance of the lead (density 1.2) of SO₄²⁻ ions attack, by adding surfactants, the green inhibitors are substances widely used in the corrosion inhibition [13-14] extracted from plants (stem of Ficus racemosa, leaves and stem of AIDS). Several studies are described in the literature concerning the green inhibitory effect.[15-17].

These can be called miracle products. They guarantee protection at low cost of extraction and nontoxic. Among the plants, we chose the Henna [18]. By using the electrochemical methods of lead in 25ml of 5M sulphuric acid, we note the ability of our inhibitor to form a protective film. [19-20]

II. Materials and methods

The lead sample was welded to a conductive wire and coated in a resin, These were mounted so that across-section area of 1.4 cm² was exposed in the electrolyte. A flat working electrode surface was obtained by mechanical polishing with emery papers 600. A platinum wire served as a counter electrode and an Hg/Hg₂SO₄ electrode was used as the reference electrode. The used electrolyte was 25 ml H₂SO₄(5M). Electrochemical measures were carried out in the absence and the presence of different concentration of surfactants. The electrochemical cell was connected to a Potentiostat/Galvanostat EC-Lab and piloted by Bio-Logic SP -200 software.

II .1 linear polarization Resistance (LPR) :

The LPR measurement involves three electrodes:

- The counter-electrode (CE) applying a polarizing current, which is usually controlled according to the lead potential or current density,
- The working electrode (WE) which is the lead sample to be analyzed,
- The reference electrode located not far from the WE surface to monitor the response of the electrochemical system to the perturbation induced by CE.

The linear polarization resistance method consists of applying to the working electrode very small voltage variations (-300mV to + 300 mV) and scanned at the rate of 1 mV.s⁻¹vs ref. above and below its corrosion potential. Over this narrow range in the vicinity of the corrosion potential the current response obtained is linear.

II .2 Cyclic polarization curves

Cyclic polarization curves were used to study the electrochemical behavior of lead in the acid sulfuric medium without and with Henna extract. The mechanism of oxidation occurs around -1.2 V and the reduction process at -0,7 V with the scanning rate of 5 mV/s.

III. Results and discussion

III .1 Tafel Curve results

To know if we chose the henna extract as a corrosion inhibitor; our first study was based on polarization curves (Tafel) of lead in the absence and in the presence of inhibitor at different concentrations at a scanning speed of potential of 1 mV/sec, allowing access to reaction rates and physical parameter values describing the state of the system.

The cathodic and anodic polarization curves of the lead electrode immersed in H₂SO₄ (5M) for 15 min for the different concentrations of inhibitor, recorded at ambient temperature, is given on figure

1 after allowing the inhibitor to react with sulphuric acid for 15 min. The values of the parameters recorded from the Tafel curves, are grouped in Table 1. These parameters are the corrosion current density (I_{corr}), the corrosion potential (E_{corr}) , the cathodic Tafel slope (b_c), the anodic Tafel slope (b_a) and the corrosion inhibitory efficiency (P_{ip}).

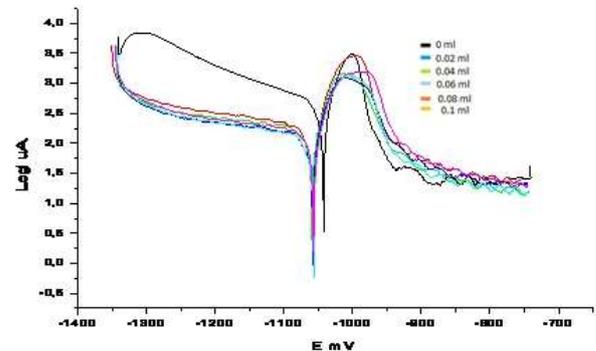


Fig1. Polarization curves of lead in H₂SO₄ without and with various Henna extracts .

The corrosion inhibitory effectiveness of lead is calculated from the load transfer strength according to relationships (1), (2):

1. From the resistance

$$P_{Rp}(\%) = (1 - R_t / R'_t) * 100 \tag{1}$$

With:

R'_t : load transfer resistance in the presence of the inhibitor;

R_t : load transfer resistance in the absence of the inhibitor.

2. From the corrosion current

$$P_i(\%) = (1 - I'_{corr} / I_{corr}) * 100 \tag{2}$$

With:

I_{corr} : the corrosion current in the absence of the inhibitor;

I'_{corr} : the corrosion current in the presence of the inhibitor.

The analysis of the results from table 1 allowed us to note that in the presence of the corrosion inhibitor:

- ❖ The corrosion current densities (I_{corr}) decrease as the Henna corrosion inhibitor concentration increases, except for V = 0.1 ml where the corrosion current has risen to 34.682 μA;
- ❖ The addition of Henna corrosion inhibitor slightly modified the E_{corr} values which became slightly more positive. Note that the effectiveness of the corrosion inhibitor increases when the concentration of the

inhibitor increases to $V = 0.08$ ml, 37.92%; the inhibition showing better inhibition ability. Besides, the decrease of I_{corr} values and the increase of P_{Rp} values with 0.08 ml also prove the studied inhibitor deprive the lead corrosion effectively [21-22].

- ❖ The addition of the Henna inhibitor to the corrosive medium results in a slight modification of the right slope of anodic

Tafel (b_a), for the volume $V = 0.02, 0.04$ and 0.06 ml;

- ❖ We notice from the above results that the best polarization resistance of lead in 5M sulphuric acid is 103 Ohm.cm^2 . It corresponds to the lowest current density of $18.034 \mu\text{A}$; for a 15 minutes immersion time after mixing the inhibitor with H_2SO_4 for 15 minutes

Table 1. Electrochemical parameters and inhibitory effectiveness of Lead corrosion in 5M H_2SO_4 , for different Henna volume.

V_{inh} (ml)	B_a (mV)	B_c (mV)	E_c (mV)	I_c (μA)	R_p (Ohm)	Efficiency P_{Rp}	Efficiency P_{Ip}
0	19.3	33.7	-1042.2	137.62	24	0	0
0.02	12.2	21	-1058.25	30.28	63.7	62.32	77.99
0.04	12.2	14,1	-1058.87	28.538	73.5	67.34	79.26
0.06	8.7	23.2	-1056.95	24.67	91.9	73.88	82.07
0.08	9.2	19.5	-1055.64	18.034	103	76.69	86.89
0.1	15.4	22	-1057.9	34.682	77.5	69.03	74.79

III .2 Adsorption and thermodynamic analysis

In order to confirm the hypothesis that the action of the inhibitor is based on a mechanism of action by simple adsorption on the surface of the metal, thus blocking the active sites and consequently, reducing the currents measured during the recording of the curves of polarization, we tried to correlate the experimental results with the plot of adsorption isotherms.

The recovery rate θ for different concentrations of Henna extract in acid medium is evaluated by the polarization curves and calculated by the relation (3):

$$\theta = (I_{corr} - I_{corrinh}) / I_{corr} \quad (3)$$

During this study, different adsorption isotherms were used:

Isotherm Langmuir adsorption

$$\theta = b V_{inh} / (1 + b V_{inh}) \quad (4)$$

Isotherm of Temkin of adsorption:

$$\text{Exp}(-2a\theta) = K V_{inh} \quad (5)$$

Isotherm of Frumkin of adsorption:

$$(\theta / 1 - \theta) \text{exp}(-2a\theta) = K V_{inh} \quad (6)$$

With:

a: is a constant of interaction between adsorbed particles;

K: is the constant balance of the adsorption process;

V_{inh} : the concentration of the inhibitor in the electrolyte.

Isotherms of adsorption calculated from Langmuir, Temkin and Frumkin are shown in (Fig.2-4). The obtained parameters are given in the (Table 2). The recovery rate θ for each concentration of henna inhibitor has been calculated and shown in the (Table 2).

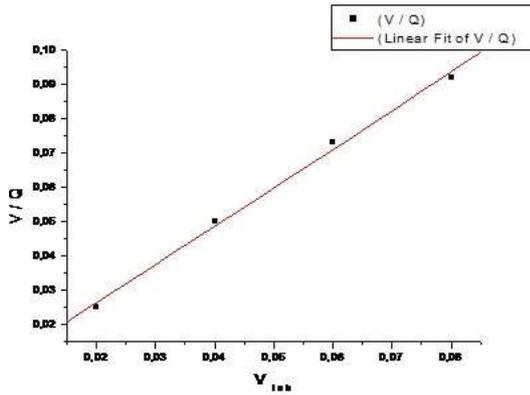


Fig 2. Langmuir adsorption isotherm of lead in H₂SO₄ in the presence of several Henna extract volumes

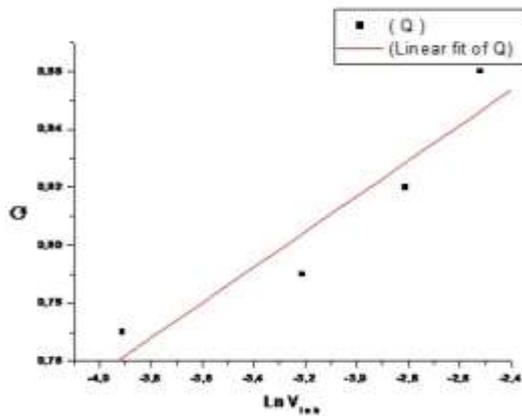


Fig 3. Temkin adsorption isotherm of lead in H₂SO₄ in the presence of several Henna extract volumes

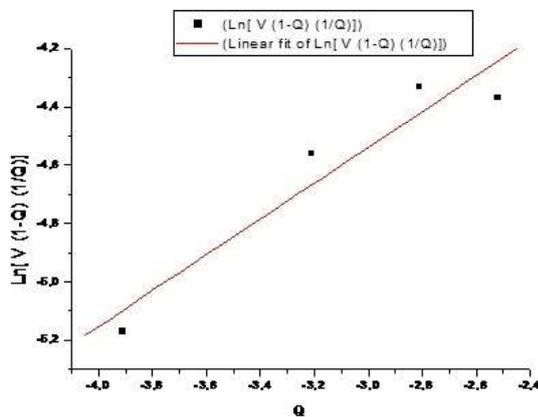


Fig 4. Frumkin adsorption isotherm of lead in H₂SO₄ in the presence of several Henna extract volumes

Table 2. Calculation of adsorption isotherm parameters of Henna extract in 5M H₂SO₄ medium.

V _{inh} (ml)	θ	V / θ	LnV	Ln V[(1- θ) / θ]
0.02	0.77	0.025	-3.91	-5.17
0.04	0.79	0.050	-3.21	-4.56
0.06	0.82	0.073	-2.81	-4.33
0.08	0.86	0.092	-2.52	-4.37
0.1	0.74	0.135	-2.30	-3.34

The correlation coefficient (R²) is an essential parameter to choose the most suitable adsorption isotherm. Usually, we select the adsorption isotherm with a correlation coefficient closing to 1. The value of correlation coefficient (R²=0.999) for Henna in curve was obtained from the Langmuir isotherm in (Fig . 2), it shows that the adsorption on the surface of the lead in H₂SO₄ medium and in the presence of Henna extract inhibitor, obeys to the adsorption isotherm of Langmuir.

As per reported literature [23], there could be two modes of adsorption of the inhibitor onto the metal surface. One is an electrostatic physisorption, whereas chemisorption is the second consequence. The Langmuir adsorption isotherm model assumes that there is a fixed number of energetically identical sites on the surface in which each of the sites can absorb only one particle of Henna. In addition, the model assumes that interactions between adsorbed particles are negligible and therefore, the adsorption energy is considered constant. With these assumptions, and for a given temperature, the relation between the adsorbed quantity of a species and its concentration in the liquid phase in contact with the surface of lead working electrode is given by equation (7).

$$\theta = b_i C_i / (1 + b_i C_i) \tag{7}$$

With:

C_i: concentration of the species i;

θ_i: surface recovery rates by species i;

b_i: adsorption coefficient.

As seen from curve of (Fig.2) the Henna extract inhibitor adsorbs according to the Langmuir isotherm. We can calculate ΔG, ΔH after determination of the equilibrium constant K relating to the formation of the protective layer.

According to the relation (4) above that done:

$$\frac{V_{inh}}{\theta} = \frac{1}{K} + V_{inh} \quad (8)$$

The slope and the intersection with the coordinate axis were been obtained from the curve of the (Figure 2).

$$\frac{V_{inh}}{\theta} = 0.004 + 1.12 V_{inh} \quad (9)$$

$$1/K = 0,004 \quad \text{then} \quad K = 250$$

The free enthalpy ΔG of adsorption is determined by the equation(10) hereafter:

$$\Delta G_{ads} = - RT \ln (55.5 * K) \quad (10)$$

Adsorption isotherms can assist in the description of the adsorption and provide information on the adsorption equilibrium and thus give a value to the free energy of adsorption [24] The value of ΔG_{ads} calculated from this equation at ambient temperature is equal to $-23.63 \text{ KJ mol}^{-1}$. The negative value of ΔG_{ads} indicates the spontaneity of the adsorption process and the stability of the adsorbed double layer on the lead surface. It is greater than -20 KJ mol^{-1} ; then it involves a charge transfer between the Henna extract inhibitor molecules and the lead surface (chemisorptions).[25-26]A. Rodríguez-Torres are obtained the related observations by evaluating plant extracts as the P. persica extract on the on AISI 1018 Carbon steel surface in $0.5 \text{ M H}_2\text{SO}_4$ is spontaneous. The ΔG_{ads}° values indicate that the adsorption process of the P. persica extract molecules is physical. Fall within the interval $-9.54 \text{ KJ.mol}^{-1} > \Delta G_{ads}^\circ < -22.45 \text{ KJ.mol}^{-1}$ at the evaluated temperatures. [27]

Calculation of adsorption heat ΔH_{ads} :

$$K = \exp \left(\frac{-\Delta H_{ads}}{RT} \right) \quad (11)$$

$$\ln K = \frac{-\Delta H_{ads}}{RT} \quad (12)$$

$$\Delta H_{ads} = -RT \ln K \quad (13)$$

The value of ΔH_{ads} calculated from the equation (13) is equal to $-13.67 \text{ kJ mol}^{-1}$. deducing that the adsorption of Henna on the lead surface is exothermic .Furthermore ,the heat adsorption Q_{ads} is defined as the opposite of the enthalpy variation ΔH_{ads} either:

$$Q_{ads} = - \Delta H_{ads} = 13.67 \text{ KJ mol}^{-1}$$

For the entropy ΔS_{ads} :

$$\Delta G_{ads} = \Delta H_{ads} - T \Delta S_{ads} \quad (14)$$

$$\Delta S_{ads} = \frac{\Delta H_{ads} - \Delta G_{ads}}{T} \quad (15)$$

The value of ΔS calculated from this equation (15) is equal to $0.033 \text{ KJ mol}^{-1} \text{ K}^{-1}$; we notice that signs of the internal terms, ΔS_{ads} and ΔH_{ads} are in opposite signs, so the variation of the temperature does not change the direction of the process.

The enthalpy (ΔH_{ads}) values are minus, which shows the adsorption is an exothermic process. The negative standard entropy ΔS_{ads} value means reduced randomness as the adsorption of inhibitor 3,3'-Diethylthiadicarbocyanine iodide on copper surface, while positive ΔS_{ads} indicates increased randomness as a replacement of water molecules by inhibitor molecules [22]. The values of ΔS_{ads} are positive in the adsorption process and this could be explained as follows:

The thermodynamic values which are obtained the algebraic sum of the adsorption of organic molecules and desorption of water molecules, therefore, the gain in enthalpy is attributed to the increase in solvent entropy [28]. The values of ΔH_{ads} provide further information about the mechanism of corrosion inhibition.

III.3 Effect of time mixing of the inhibitor:

A Three volumes of acidic medium H_2SO_4 5 M and 0.08 ml of Henna extract were been mixed respectively during 15.30 and 45 minutes. These mixtures were introduced separately in the electrochemical cell and after a waiting time of 15 minutes. The Tafel plot was realized and the obtained curves are shown in the (Fig. 5). The characteristic parameters obtained from curves of the (Fig.5) are given in (Table 3). The used scan rate of the potential was 1 mV/s.

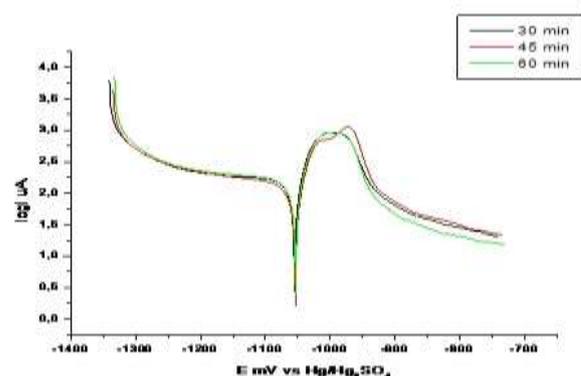


Fig 5. Polarization curves of lead in H_2SO_4 5M and 0.08ml of Henna extract at different mixing time with inhibitor.

Table 3. Electrochemical parameters of lead corrosion in 5M H₂SO₄ and 0.08ml of Henna extract

Time of mixture (mn)	B _a (mV)	B _c (mV)	E _c (mV)	I _c (μA)	R _p (Ohm)
15	13.5	29.4	-1056.50	27.23	92.6
30	9.1	11.8	-1054.38	14.734	109
45	12.5	17.7	-1054.44	19.26	102

From the results shown in (Table 3) it is noted that the time of mixture gives a slight variation in the corrosion resistance. In the inhibited systems did not change to various temps (30 –45 min) relatively around the same value of R_p . These observations show that the inhibition of lead corrosion by Henna was able to protect despite the acidity H₂SO₄ 5M .The good mixing time for inhibitor with sulphuric acid corresponds to 109 Ohm.cm² for 30 min corresponding to a decrease in current densities (14.73μA/cm²) .The adsorbed inhibitor molecules block the surface active sites and decrease the area available for hydrogen evolution and metal dissolution reactions[29].

a)Variation in immersion time:

To deepen the study of the inhibitor, we plotted the corrosion current curves and the corrosion resistance versus the immersion time; after setting the time of the inhibiting mixture with 5M sulphuric acid at 30mn. Fig. 6-9 show the lead polarization curves obtained in a solution containing 5 M H₂SO₄ and 0.08 ml Henna extract. All curves were plotted from the cathodic domain to the anode domain with a scan rate of 1mV/s.(Table 4) groups the electrochemical parameters resulting from the lead polarization curves in the presence and in the absence of 0.08 ml of Henna extract.

❖ 15min

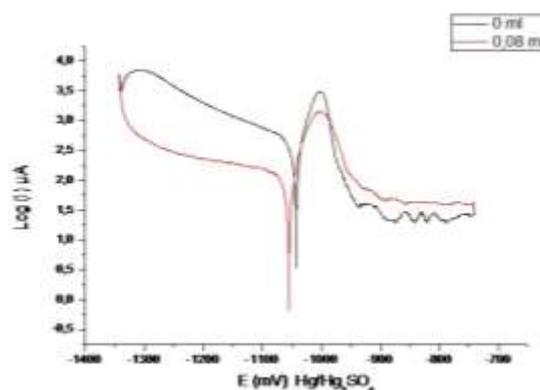


Fig 6. Polarization curves of lead in H₂SO₄ in the Presence and in the absence of 0.08 ml of Henna extract.

❖ 30 min

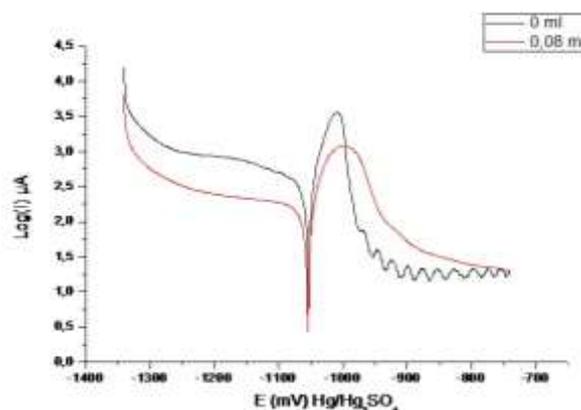


Fig 7. Polarization curves of lead in H₂SO₄ in the presence and in the absence of 0.08 ml of Henna extract.

❖ 45 min

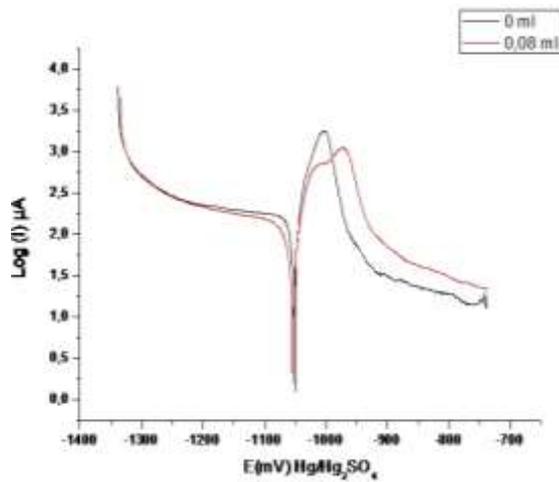


Fig 8. Polarization curves of lead in H_2SO_4 in the presence and the absence of 0.08 ml of Henna extract.

❖ 60 min

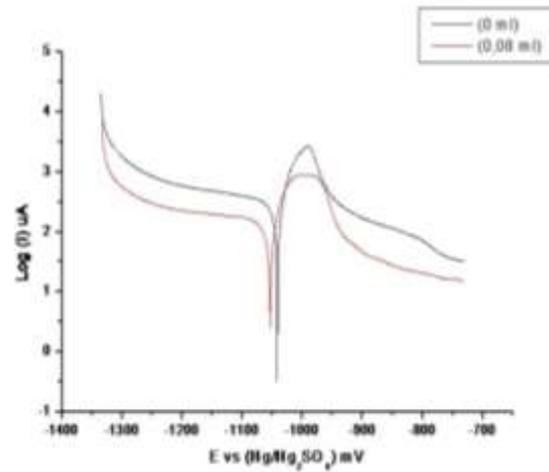


Fig 9. Polarization curves of lead in H_2SO_4 in the presence and in the absence of 0.08 ml of Henna extract

Table 4. Electrochemical parameters of the corrosion of lead with and without 0.08 ml of Henna extract in H_2SO_4 5M.

Time of immersion (min)	$V_{(Inh)}$ ml	B_a (mV)	B_c (mV)	E_c (mV)	I_c (μA)	E%	R_p (Ohm)
15	0	5.6	6.8	-1042.22	34.233	-	30
	0.08	9.6	16.4	-1053.77	17.44	49.05	95.6
30	0	12.3	6.8	-1052.69	88.233	-	24.7
	0.08	6.7	8.9	-1054.81	13.9	84.24	98.2
45	0	12.8	25.3	-1050.13	43.16	-	48.2
	0.08	11.8	18.1	-1053.41	20.47	52.57	116
60	0	8.2	16.0	-1040.65	44.751	-	29.2
	0.08	11.4	18.5	-1041.14	54.84	-	72.5

According to the results of Table 4 it was noted that the time of immersion of 30 minutes gives the best performance for henna extract in the acidic medium regarding to the current density. The calculated efficiency is equal 84.24 %. Furthermore, the adsorption pulp of Jujube extracts on the metal surface, which leads to the blocking of the active sites and reduction the attack of the chloride ions at the metal-acid interface [30-33].

IV. Conclusion

The objectives of our work were to study the effectiveness of the Henna extract in the sulphuric acid medium. Electrochemical properties were studied by Tafel plot polarisation a function of mixing time and immersion. A volume of 0.08 ml Henna extracts in 25ml H₂SO₄ was able to form a protective layer on the surface of lead electrode. The best efficiency of 84.24 % is obtained at the immersion of 30 minutes. The molecule of Henna extract was adsorbed on the active sites of the lead surface according to the Langmuir isotherm.

The adsorption is spontaneous regarding the value of ΔG_{ads} that is negative. From ΔS_{ads} and ΔH_{ads} those are in opposite signs, so the variation of the temperature does not have an effect on the direction of the process of protection by the Henna extract.

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